

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Commissioner
US Department of Commerce
United States Patent and Trademark
Office, PCT
2011 South Clark Place Room
CP2/5C24
Arlington, VA 22202
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 09 October 2001 (09.10.01)	
International application No. PCT/US00/05458	Applicant's or agent's file reference M 6712 HST/NI
International filing date (day/month/year) 02 March 2000 (02.03.00)	Priority date (day/month/year) 02 March 1999 (02.03.99)
Applicant KAWAGUCHI, Jun et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

23 June 2000 (23.06.00)

☐ in a notice effecting later election filed with the International Bureau on:2. The election ☒ was☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland	Authorized officer Elisabeth KÖNIG
Facsimile No.: (41-22) 740.14.35	Telephone No.: (41-22) 338.83.38

INTERNATION SEARCH REPORT

International application No.
PCT/US00/05458

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) :C23C 22/07

US CL :148/262

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 148/262, 148/241; 205/111, 318, 189

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EAST

search terms: zinc phosphate, phosphoric acid, nitric acid, sludge, sludging, nonsludging

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,525,431 A (KANAMARU et al) 11 June 1996, col. 3, lines 14-40; col. 15, lines 25-55; col. 18, lines 19-51; col. 20, lines 29-32 and 63-68; col. 21, lines 1-2; col. 22, lines 31-35.	1-10
Y	US 5,503,733 A (SPECKMANN et al) 02 April 1996, abstract, col. 2, lines 50-63 ; col. 3, lines 45-50; col. 4, lines 29-33 and 47-49; col 5, lines 16-42; claim 1.	1-11
Y	US 5,203,930 A (BLUMLHUBER et al) 20 April 1993, abstract, col. 2, lines 25-30; col. 3, lines 17-27; claim 1.	1-7

☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:	*T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
A document defining the general state of the art which is not considered to be of particular relevance	*X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
E earlier document published on or after the international filing date	*Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
L document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	* & * document member of the same patent family
O document referring to an oral disclosure, use, exhibition or other means	
P document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

26 APRIL 2000

Date of mailing of the international search report

16 MAY 2000

Name and mailing address of the ISA/US
Commissioner of Patents and Trademarks
Box PCT
Washington, D.C. 20231

Facsimile No. (703) 305-3230

Authorized officer

ANDREW L OLTMANS

Telephone No. (703) 308-0661

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/05458

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4,950,339 A (GEHMECKER et al) 21 <i>August 1990</i> abstract; col. 1, lines 63-68; col. 2, lines 1-4 and 51 and 65; col. 3, lines 46-52, claim 1.	1-7
Y	US 3,647,568 A (LARSON) 07 March 1972, col. 2, lines 30-50.	1-7

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PATENT COOPERATION TREATY

PCT

REC'D 25 MAR 2002

WIPO PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference M 6712 HST/N		FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/US00/05458	International filing date (day/month/year) 02 March 2000 (02.03.2000)	Priority date (day/month/year) 02 March 1999 (02.03.1999)	
International Patent Classification (IPC) or national classification and IPC IPC(7): C23C 22/07 and US Cl.: 148/262			
Applicant HENKEL CORPORATION			
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of <u>5</u> sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of <u>0</u> sheets.</p>			
<p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the report</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of report with regard to novelty, inventive step and industrial applicability</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input type="checkbox"/> Certain observations on the international application</p>			
Date of submission of the demand 23 June 2000 (23.06.2000)		Date of completion of this report 08 March 2002 (08.03.2002)	
Name and mailing address of the IPEA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703)305-3230		Authorized officer Andrew L Oltmans Jean Proctor Paralegal Specialist Telephone No. 703-308-0661	

Form PCT/IPEA/409 (cover sheet)(July 1998)

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.

PCT/US00/05458

I. Basis of the report

1. With regard to the elements of the international application:*



the international application as originally filed.



the description:

pages 1-11 as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of _____.



the claims:

pages 11 and 12, as originally filed

pages NONE, as amended (together with any statement) under Article 19

pages NONE, filed with the demand

pages NONE, filed with the letter of _____.



the drawings:

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of _____.



the sequence listing part of the description:

pages NONE, as originally filed

pages NONE, filed with the demand

pages NONE, filed with the letter of _____.

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language _____ which is:



the language of a translation furnished for the purposes of international search (under Rule 23.1(b)).



the language of publication of the international application (under Rule 48.3(b)).



the language of the translation furnished for the purposes of international preliminary examination (under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:



contained in the international application in printed form.



filed together with the international application in computer readable form.



furnished subsequently to this Authority in written form.



furnished subsequently to this Authority in computer readable form.



The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.



The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☐ The amendments have resulted in the cancellation of:



the description, pages NONE



the claims, Nos. NONE



the drawings, sheets/fig NONE

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).**

* Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17).

** Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US00/05458

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. STATEMENT

Novelty (N)	Claims <u>1-11</u>	YES
	Claims <u>NONE</u>	NO
Inventive Step (IS)	Claims <u>NONE</u>	YES
	Claims <u>1-11</u>	NO
Industrial Applicability (IA)	Claims <u>1-11</u>	YES
	Claims <u>NONE</u>	NO

2. CITATIONS AND EXPLANATIONS

Please See Continuation Sheet

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US00/05458

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

LACK OF INVENTIVE STEP

SPECKMANN et al. U.S. Patent No. 5,703,733

Claims 1-11 lack an inventive step under PCT Article 33(3) as being obvious over SPECKMANN et al. U.S. Patent No. 5,703,733 (SPECKMANN).

SPECKMANN teaches a zinc phosphating solution having the following composition, which overlaps the composition, including the compositional equations, recited in instant claims 1-6 (column 2):

- 50 a) phosphating solutions containing the following components are used:
- Zn²⁺ cations in quantities of 0.1 to 5 g/l,
 - PO₄³⁻ anions in quantities of 5 to 50 g/l,
 - NO₃⁻ anions in quantities of 0.1 to 50 g/l and
 - 55 Mn²⁺ cations in quantities of 0.1 to 5 g/l and
 - Cu²⁺ cations in quantities of 0.001 to 1 g/l,

SPECKMANN also teaches the addition of fluorine compounds, as recited in instant claim 7 (column 4, lines 47-49). SPECKMANN also teaches the process of coating metal substrates wherein the conditions overlap those recited in instant claims 8-10 (column 2):

- 60 b) the following conditions are established: pH value of the phosphating solutions 1.5 to 4.5, temperature of the phosphating solutions 10° to 80° C., treatment time 1 to 300 seconds,
- c) the workpieces are cathodically treated during phosphating with a direct current having a density of 0.01 to 100 mA/cm².

[emphasis added by examiner]

SPECKMANN teaches that the process includes contacting the liquid composition with a counter electrode and causing an electric current to flow through the metal substrate and into the volume of the liquid composition, as recited in instant claim 8 (column 3, lines 45-50). SPECKMANN further teaches a pretreatment with the composition recited in instant claim 11 (column 5, lines 34-41 and

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No.
PCT/US00/05458

Supplemental Box

(To be used when the space in any of the preceding boxes is not sufficient)

column 7, lines 26-30).

SPECKMANN fails to meet all of the limitations of the instant claims in that SPECKMANN fails to explicitly teach the exact compositional and process condition ranges instantly claimed.

However, one of ordinary skill in the art would find the instant invention obvious because the compositional ranges and the process conditions taught in SPECKMANN overlap the compositional ranges recited in the instant claims.

KANAMARU et al. U.S. Patent No. 5,525,431

Claims 1-10 lack an inventive step under PCT Article 33(3) as being obvious over KANAMARU et al. U.S. Patent No. 5,525,431 (KANAMARU).

KANAMARU teaches an electrolyte composition for electrolytically coating metal substrates wherein the electrolyte contains phosphoric acid, nitric acid and dissolved zinc cations in a concentration that overlaps the concentration, including the compositional equations, recited in instant claims 1-7 (column 15):

Such oxide film can be prepared, for example, by dipping zinc-base galvanized sheet steel in an aqueous solution containing 1-70 g/l of potassium permanganate, 5-60 g/l of phosphoric acid or boric acid (when the two acids are used together, respectively 5-60 g/l) and 100-800 g/l of zinc nitrate, by subjecting the galvanized sheet steel to a cathodic electrolytic treatment in said aqueous solution, or by spraying the aqueous solution onto the galvanized sheet steel, whereby Mn oxide, phosphoric acid and Zn oxide are formed simultaneously.

[emphasis added by examiner]
and

An etching agent, for example, at least one of sulfuric acid, nitric acid, perchloric acid, etc. is preferably added to the above-mentioned aqueous solution in an amount of 1-10 g/l to improve the adhesive property, etc. of the film.

[emphasis added by examiner]

(see also column 18, lines 19-51)

KANAMARU also teaches a process of coating metal substrates wherein the process conditions overlap those recited in the instant claims (column 18, line 25 and 50). KANAMARU teaches that the process includes contacting the liquid composition with a counter electrode and causing an electric current to flow through the metal substrate and into the volume of the liquid composition, as recited in instant claim 8 (column 20, line 63 to column 23, line 3 and column 22, lines 28-35).

KANAMARU fails to meet all of the limitations of the instant claims in that KANAMARU fails to explicitly teach the exact compositional and process condition ranges instantly claimed.

However, one of ordinary skill in the art would find the instant invention obvious because the compositional ranges and the process conditions taught in KANAMARU overlap the compositional ranges recited in the instant claims.

INDUSTRIAL APPLICABILITY

Claims 1-11 meet the requirement as defined by PCT Article 33(2) and 33(4) because the liquid composition and the process for forming a zinc phosphate conversion coating find use in the metal finishing industry.

----- NEW CITATIONS -----

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

To: HARPER, STEPHEN D.
HENKEL CORPORATION
2500 RENAISSANCE BOULEVARD
SUITE 200
GULPH MILLS, PA 19406

US
Rev IDS/ISR - 6/16/00
Rmdr IDS/ISR - 7/16/00
File IDS/ISR - 8/16/00

Ind
Rev Search Rpt PCT - 6/16/00
PCT

NOTIFICATION OF TRANSMITTAL OF THE INTERNATIONAL SEARCH REPORT OR THE DECLARATION

(PCT Rule 44.1)

Applicant's or agent's file reference M 6712 HST/NI	Date of Mailing (day/month/year) 16 MAY 2000
International application No. PCT/US00/05458	International filing date (day/month/year) 02 MARCH 2000
Applicant HENKEL CORPORATION	

1. ☒ The applicant is hereby notified that the international search report has been established and is transmitted herewith.

Filing of amendments and statement under Article 19:

The applicant is entitled, if he so wishes, to amend the claims of the international application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the international search report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No.: (41-22) 740.14.35

ACTION _____ INFO COB

REC'D MAY 17 2000

For more detailed instructions, see the notes on the accompanying sheet.

DUE _____

FILE M 6712 HST/NI

2. ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.

c: US Search Rpts.

3. ☐ With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

- ☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.
- ☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Further action(s):** The applicant is reminded of the following:

Shortly after 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in rules 90 bis 1 and 90 bis 3, respectively, before the completion of the technical preparations for international publication.

Within 19 months from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).

Within 20 months from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer <i>Andrew L Oltmans</i> ANDREW L OLTMANS Telephone No. (703) 308-0661
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PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference M 6712 HST/NI	FOR FURTHER ACTION	see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.
International application No. PCT/US00/05458	International filing date (day/month/year) 02 MARCH 2000	(Earliest) Priority Date (day/month/year) 02 MARCH 1999
Applicant HENKEL CORPORATION		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

- a. With regard to the language, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
- b. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international search was carried out on the basis of the sequence listing:
- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.
2. ☐ Certain claims were found unsearchable (See Box I).
3. ☐ Unity of invention is lacking (See Box II).
4. With regard to the title,
- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established by this Authority to read as follows:
5. With regard to the abstract,
- ☒ the text is approved as submitted by the applicant.
- ☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.
6. The figure of the drawings to be published with the abstract is Figure No. _____
- ☐ as suggested by the applicant.
- ☐ because the applicant failed to suggest a figure.
- ☐ because this figure better characterizes the invention.
- ☐ None of the figures.

NOTES TO FORM PCT/ISA/220 (continued)

The following examples illustrate the manner in which amendments must be explained in the accompanying letter:

1. [Where originally there were 48 claims and after amendment of some claims there are 51]:
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."
2. [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."
3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged; claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."
4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged; claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

"Statement under Article 19(1)" (Rule 46.4)

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

The statement should be brief, it should not exceed 500 words if in English or if translated into English.

It should not be confounded with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It should not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

In what language?

The amendments must be made in the language in which the international application is published. The letter and any statement accompanying the amendments must be in the same language as the international application if that language is English or French; otherwise, it must be in English or French, at the choice of the applicant.

Consequence if a demand for international preliminary examination has already been filed?

If, at the time of filing any amendments under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the same time of filing the amendments with the International Bureau, also file a copy of such amendments with the International Preliminary Examining Authority (see Rule 62.2(a), first sentence).

Consequence with regard to translation of the international application for entry into the national phase?

The applicant's attention is drawn to the fact that, where upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see Volume II of the PCT Applicant's Guide.

H3PO4
 98 g/mol
 x
 0.2 mol/L
 19.6 g/L

Zn
 65.38 g/mol
 x
 0.144 mol/L
 9.41472 g/L

nitric acid
 63.02 g/mol
 x
 0.2 mol/L
 12.604 g/L

Total Equation

Input
 H3PO4 nitric acid
 0.2 0.2

Calculated Zn conc
 H3PO4 nitric acid
 upper limit 0.06 0.1 0.16
 lower limit 0.054 0.09 0.144

Zn
 65.38 g/mol
 x
 upper limit 0.16 mol/L 10.4608 g/L
 lower limit 0.144 mol/L 9.41472 g/L

H3PO4	
98 g/mol	
x	
0.6 mol/L	58.8 g/L
Zn	
65.38 g/mol	
x	
0.144 mol/L	9.41472 g/L
nitric acid	
63.02 g/mol	
x	
0.6 mol/L	37.812 g/L

Total Equation			
Input			
H3PO4	0.6	nitric acid	
Calculated Zn conc			
H3PO4	0.18	0.3	0.48
upper limit			
lower limit	0.162	0.27	0.432
Zn			
65.38 g/mol			
x			
upper limit	0.48 mol/L		31.3824 g/L
lower limit	0.432 mol/L		28.24416 g/L

cess of this invention enables zinc phosphate treatment to be run very rapidly through the use of electrolysis. This feature, in combination with the fact that this process can be used to execute zinc phosphate treatment on essentially any material that is electrically conductive, makes the instant process highly advantageous on an industrial or commercial basis.

CLAIMS

1. A liquid composition of matter that is suitable as electrolyte for a nonsludging electrolytic zinc phosphate treatment process, said liquid composition comprising water, dissolved phosphoric acid, dissolved nitric acid, dissolved zinc cations, m chemically distinct species of cations other than zinc, and n chemically distinct species of anions other than anions derivable by ionization of phosphoric and nitric acids, each of m and n independently being zero or a positive integer, the concentration of zinc in moles per liter in said liquid composition satisfying the following mathematical condition:

$$\{Zn\} \leq 0.3 \{H_3PO_4\} + 0.5 \{HNO_3\} - 0.5 \sum_{i=0}^m p_i C_i + 0.5 \sum_{j=0}^n q_j A_j$$

in which: " $\{Zn\}$ ", " $\{H_3PO_4\}$ ", and " $\{HNO_3\}$ " respectively represent the zinc, phosphoric acid, and nitric acid concentrations in mol/L; each of C_0 and A_0 is zero; each of p_0 and q_0 is 1; if m is not zero, for each positive integer i from 1 to m , C_i represents the concentration in mol/L of the i th distinct cation species other than zinc present in the bath and p_i represents the cationic valence of said i th distinct cation species; and if n is not zero, for each positive integer j from 1 to n , A_j represents the concentration in mol/L of the j th distinct anion species other than anions derivable by ionization of phosphoric or nitric acids present in the bath and q_j represents the anionic valence of said j th distinct anion species.

2. A liquid composition according to claim 1, wherein:

- the phosphoric acid concentration is from 0.10 to 0.60 mol/L;
- the nitric acid concentration is from 0.20 to 1.0 mol/L; and

$$\{Zn\} \geq 0.15 \{H_3PO_4\} + 0.25 \{HNO_3\} - 0.25 \sum_{i=0}^m p_i C_i + 0.25 \sum_{j=0}^n q_j A_j$$

3. A liquid composition according to claim 2, wherein:

- the phosphoric acid concentration is from 0.25 to 0.50 mol/L;
- the nitric acid concentration is from 0.65 to 0.90 mol/L; and

$$\{Zn\} \geq 0.27 \{H_3PO_4\} + 0.45 \{HNO_3\} - 0.45 \sum_{i=0}^m p_i C_i + 0.45 \sum_{j=0}^n q_j A_j$$

4. A liquid composition according to claim 3, wherein $\{Zn\}/\{H_3PO_4\} < 0.91$.

5. A liquid composition according to claim 2, wherein $\{Zn\}/\{H_3PO_4\} < 0.91$.
6. A liquid composition according to claim 1, wherein $\{Zn\}/\{H_3PO_4\} < 0.91$.
7. A liquid composition according to any one of claims 1 through 6, additionally comprising at least one additive selected from the group consisting of nitrous acid, permanganic acid, peroxy-sulfuric acid, hydrogen peroxide, chloric acid, perchloric acid, nitrobenzene sulfonic acid, hydroxylamine, starch/phosphoric acid esters, fluorine compounds, and salts of all of the other materials previously recited in this group for which salts are known.
8. A process for forming a zinc phosphate conversion coating on a metal substrate without generating any sludge thereby, said process comprising operations of:
- (I) bringing said metal substrate into contact with a volume of a liquid composition according to any one of claims 1 through 7, said volume of liquid composition also being in contact with a counter electrode that is distinct from said metal substrate; and
- (II) causing electric current to flow in a cathodizing direction through said metal substrate into said volume of liquid composition and through said counter electrode.
9. A process according to claim 8, wherein:
- said volume of liquid composition is maintained during operation (II) at a temperature that is between 50 and 85 °C; and
 - in operation (II) there is a current density through said metal substrate that is between 0.5 and 50 A/dm².
10. A process according to claim 9, wherein:
- said volume of liquid composition is maintained during operation (II) at a temperature that is between 75 and 85 °C; and
 - in operation (II) there is a current density through said metal substrate that is between 7.0 and 15 A/dm².
11. A process according to any one of claims 8 through 10, wherein prior to operation (I), said metal substrate is brought into contact with a weakly basic aqueous colloidal solution that contains titanium oxide, titanium hydroxide, and zinc phosphate.

Preliminary Amendment at Entry into the U. S. National Stage of International Application
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IN THE CLAIMS:

Amend claims 7, 8 and 11 to read as follows:

- A3
7. (Amended) A liquid composition according to claim 1, additionally comprising at least one additive selected from the group consisting of nitrous acid, permanganic acid, peroxysulfuric acid, hydrogen peroxide, chloric acid, perchloric acid, nitrobenzene sulfonic acid, hydroxylamine, starch/phosphoric acid esters, fluorine compounds, and salts of all of the other materials previously recited in this group for which salts are known.
8. (Amended) A process for forming a zinc phosphate conversion coating on a metal substrate without generating any sludge thereby, said process comprising operations of:
- (I) bringing said metal substrate into contact with a volume of a liquid composition according to claim 1, said volume of liquid composition also being in contact with a counter electrode that is distinct from said metal substrate; and
 - (II) causing electric current in flow in a cathodizing direction through said metal substrate into said volume of liquid composition and through said counter electrode.
11. (Amended) A process according to claim 8, wherein prior to operation (I), said metal substrate is brought into contact with a weakly basic aqueous colloidal solution that contains titanium oxide, titanium hydroxide, and zinc phosphate.
- A4

Enter new claims 12-21 as follows:

- A5
- 12. (New) A liquid composition that is suitable as electrolyte for a nonsludging electrolytic zinc phosphate treatment process, said liquid composition comprising water, at least 0.10 mol/L dissolved phosphoric acid, at least 0.10 mol/L dissolved nitric acid, dissolved zinc cations, m chemically distinct species of cations other than zinc, and n chemically distinct species of anions other than anions derivable

by ionization of phosphoric and nitric acids, each of m and n independently being zero or a positive integer, the concentration of zinc in moles per liter in said liquid composition satisfying both of the following mathematical conditions:

$$\{Zn\} \leq 0.3 \{H_3PO_4\} + 0.5 \{HNO_3\} - 0.5 \sum_{i=0}^m p_i C_i + 0.5 \sum_{j=0}^n q_j A_j; \text{ and}$$

$$\{Zn\} \geq 0.15 \{H_3PO_4\} + 0.25 \{HNO_3\} - 0.25 \sum_{i=0}^m p_i C_i + 0.25 \sum_{j=0}^n q_j A_j;$$

in which : " $\{Zn\}$ ", " $\{H_3PO_4\}$ ", and " $\{HNO_3\}$ " respectively represent the zinc, phosphoric acid, and nitric acid concentrations in mol/L; each of C_0 and A_0 is zero; each p_0 and q_0 is 1; if m is not zero for each positive integer j from 1 to m , C_j represents the concentration in mol/L of the j th distinct cation species other than zinc present in the bath and p_j represents the cationic valence of said j th distinct cation species; and if n is not zero, for each positive integer j from 1 to n , A_j represents the concentration in mol/L of the j th distinct anion species other than anions derivable by ionization of phosphoric or nitric acids present in the bath and q_j represents the anionic valence of said j th distinct anion species, wherein $\{Zn\}/\{H_3PO_4\} < 0.91$.--

--13. (New) A liquid composition according to claim 12, additionally comprising 0.0005 to 1.0 mol/L of at least one additive selected from the group consisting of nitrous acid, permanganic acid, peroxysulfuric acid, hydrogen peroxide, chloric acid, perchloric acid, nitrobenzene sulfonic acid, hydroxylamine, starch/phosphoric acid esters, fluorine compounds, and salts of all the other materials previously recited in this group for which salts are known.--

--14. (New) A liquid composition of matter that is suitable as electrolyte for a nonsludging electrolytic zinc phosphate treatment process, said liquid composition comprising water, at least 0.20 mol/L dissolved

phosphoric acid, at least 0.20 mol/L dissolved nitric acid, dissolved zinc cations, m chemically distinct species of cations other than zinc, and n chemically distinct species of anions other than anions derivable by ionization of phosphoric and nitric acids, each of m and n independently being zero or a positive integer, the concentration of zinc in moles per liter in said liquid composition satisfying both of the following mathematical conditions:

$$\{Zn\} \leq 0.3 \{H_3PO_4\} + 0.5 \{HNO_3\} - 0.5 \sum_{i=0}^m p_i C_i + 0.5 \sum_{j=0}^n q_j A_j; \text{ and}$$

$$\{Zn\} \geq 0.27 \{H_3PO_4\} + 0.45 \{HNO_3\} - 0.45 \sum_{i=0}^m p_i C_i + 0.45 \sum_{j=0}^n q_j A_j;$$

in which : “ $\{Zn\}$ ”, “ $\{H_3PO_4\}$ ”, and “ $\{HNO_3\}$ ” respectively represent the zinc, phosphoric acid, and nitric acid concentrations in mol/L; each of C_0 and A_0 is zero; each p_0 and q_0 is 1; if m is not zero for each positive integer j from 1 to m , C_j represents the concentration in mol/L of the j th distinct cation species other than zinc present in the bath and p_j represents the cationic valence of said j th distinct cation species; and if n is not zero, for each positive integer j from 1 to n , A_j represents the concentration in mol/L of the j th distinct anion species other than anions derivable by ionization of phosphoric or nitric acids present in the bath and q_j represents the anionic valence of said j th distinct anion species, wherein $\{Zn\}/\{H_3PO_4\} < 0.91$.--

--15. (New) A liquid composition according to claim 14, additionally comprising 0.0005 to 1.0 mol/L of at least one additive selected from the group consisting of nitrous acid, permanganic acid, peroxysulfuric acid, hydrogen peroxide, chloric acid, perchloric acid, nitrobenzene sulfonic acid, hydroxylamine, starch/phosphoric acid esters, fluorine compounds, and salts of all the other materials previously recited in this group for which salts are known--

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--16. (New) A process for forming a zinc phosphate conversion coating on a metal substrate without generating any sludge thereby, said process comprising operations of:

- AS
Contd.
- (I) bringing said metal substrate into contact with a volume of a liquid composition according to claim 12, said volume of liquid composition also being in contact with a counter electrode that is distinct from said metal substrate; and
 - (II) causing electric current to flow in a cathodizing direction through said metal substrate into said volume of liquid composition and through said counter electrode--

--17. (New) A process according to claim 16, wherein:

- said volume of liquid composition is maintained during operation (II) at a temperature that is between 50 and 85°C; and
- in operation (II) there is a current density through said metal substrate that is between 0.5 and 50 A/dm².--


--18. (New) A process according to claim 16, wherein prior to operation (I), said metal substrate is brought into contact with a weakly basic aqueous colloidal solution that contains titanium oxide, titanium hydroxide, and zinc phosphate--

--19. (New) A process for forming a zinc phosphate conversion coating on a metal substrate without generating any sludge thereby, said process comprising operations of:

- (I) bringing said metal substrate into contact with a volume of a liquid composition according to claim 14, said volume of liquid composition also being in contact with a counter electrode that is distinct from said metal substrate; and
- (II) causing electric current to flow in a cathodizing direction through said metal substrate into said volume of liquid composition and through said counter electrode.--

--20. (New) A process according to claim 19, wherein:

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- said volume of liquid composition is maintained during operation (II) at a temperature that is between 75 and 85°C; and
 - in operation (II) there is a current density through said metal substrate that is between 7.0 and 15 A/dm².--

--21. (New) A process according to claim 19, wherein prior to operation (I), said metal substrate is brought into contact with a weakly basic aqueous colloidal solution that contains titanium oxide, titanium hydroxide, and zinc phosphate.--

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